#### NATIONAL BUREAU OF STANDARDS REPORT

10 064

Progress Report

on

PLASTIC REINFORCED EBA CEMENTS
AS TEMPORARY RESTORATIVE MATERIALS



U.S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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#### NATIONAL BUREAU OF STANDARDS REPORT

**NBS PROJECT** 

**NBS REPORT** 

311.05-11-3110560

June 30, 1969

10 064

Progress Report

on

#### PLASTIC REINFORCED EBA CEMENTS AS TEMPORARY RESTORATIVE MATERIALS

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This investigation is part of the dental research program conducted by the National Bureau of Standards and is supported by the Dental Research Division of the U.S. Army Medical Research and Development Command.

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### Plastic Reinforced EBA Cements as Temporary Restorative Materials

Previous investigations have resulted in the development of 1-5 improved zinc oxide-eugenol-o-ethoxybenzoic acid (EBA) cements.

These materials have been well accepted for use as luting media for fixed restorations and as insulating bases. The brittleness of these cements, however, has limited their use for the temporary restoration of multiple surface carious lesions in areas subject to heavy masticatory forces. The purpose of this study was to investigate the possibility of improving the stress-bearing characteristics of ZOE-EBA cements through the incorporation of powdered polymers of relatively low elastic moduli.

#### Materials\* and Methods

\* Certain commercial materials and equipment are identified in this report to adequately specify the experimental procedure. In no instance does such identification imply recommendation or endorsement by the National Bureau of Standards; neither does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Zinc oxide, reagent grade (unless otherwise indicated) was passed through a number 70 sieve. Zinc oxides of varying particle size, carbon dioxide and water content were prepared from a base oxide that fulfilled U.S.P. requirements T. Tabular alumina had a particle size ranging from < 1 µm to > 20 µm with few particles > 20 µm. This aluminum oxide was silanized for use in one experimental formulation.

The material (64 gm) was slurried with acetone. To this slurry 3-methacryloxypropyl tri(methoxy)silane (12 ml) was added.

The solvent was evaporated on a steam bath and the product dried in an oven at 110°C for 8 hours. The aluminum oxide was also surface coated with varying amounts of polymer by evaporation of acetone solutions containing 3.0, 0.5, and 0.05 percent methyl methacrylate copolymer.

A highly dispersed gamma aluminum oxide with particle size diameter

<sup>+</sup> Baker and Adamson, Allied Chemical Co., New York, N. Y.

T The New Jersey Zinc Co., Palmerton, Pa.

Φ T-61 Tabular Alumina, Aluminum Company of America, Bauxite, Ark.

<sup>§</sup> A-174 Silane, Union Carbide Corp., New York, N. Y.

<sup>¶</sup> Acryloid K-120N, Rohm and Haas, Philadelphia, Pa.

Il Aluminum Oxide C, Degussa Inc. Kearny, N. J.

of 5 to 30 µm and a surface area of 5 to 30 m²/g which had been manufactured by flame hydrolysis of aluminum chloride was employed in one formulation. Spherical aluminum oxide was 1-20 µm particle size. Crushed hydrogenated rosin and a terpene resin were passed through a number 100 sieve. Bentonite coated wood rosin was technical grade. The sources of the various polymers are given in Table 1. The aluminum sulfate, EBA and eugenol were reagent the physical properties of available grade. Two commercially/temporary restorative materials (cement A and cement B and cement B s were also evaluated.

<sup>#</sup> Federal Mogul Division, Ann Arbor, Mich.

<sup>\*\*</sup> Staybelite, Hercules, Inc., Wilmington, Del.

<sup>++</sup> Piccolite Alpha - 115, Pennsylvania Industrial Chemical Corp., Clairton, Pa.

TT Penresina, S. B. Penick and Co., New York, N. Y.

 $<sup>\</sup>Phi\Phi$  B and T Cement, The L. D. Caulk Co., Milford, Del.

<sup>§§</sup> Fynal, The L. D. Caulk Co., Milford, Del.

Unless otherwise stated, the powder components were prepared by thorough mixing of the various components. The composition of the liquid was 62.5 percent EBA and 37.5 percent eugenol. In preparing the cements, a powder-liquid ratio was selected which, after thorough mixing for 1-2 minutes, gave a consistency suitable for clinical use. Setting times, compressive strengths and solubility and disintegration were determined by the procedures prescribed by American Dental Association Specification No. 8.6 Headspeed of the Universal Testing Machine for the compressive strength measurements was 0.035 in/min. Besides one day storage tests, some additional data were obtained on storage for one week. Tensile strengths of specimens, after conditioning similar to that employed for compressive strength measurements, were measured by the diametral method using specimens 12 mm in length and 6 mm in diameter. Headspeed of the Universal Testing Machine was 0.2 in/min. The coefficients of variation (c.v.) were calculated from the range of values obtained using the formula:

c.v. = range  $\cdot$  100/(d<sub>n</sub>  $\cdot$   $\bar{x}$ )

where  $d_n$  is approximately equal to  $\sqrt{n}$  for 3  $\leq$  n 10 and  $\bar{x}$  is the average value. The values for  $d_n$  were taken from Natrella.<sup>8</sup>

In a limited clinical study, approximately 50 restorations, including complex restorations subject to heavy occlusal stresses, were placed using one formulation. Its powder component contained 58.2 percent ZnO, 27.3 percent Al<sub>2</sub>O<sub>3</sub>, 5.4 percent rosin and 9.1 percent methyl methacrylate copolymer. A powder-liquid ratio of 1.2 gm powder per 0.2 ml of liquid was used. The material was usually mixed on a glass slab, but could also be mixed by employing a mechanical mixer. The unreacted eugenol was removed by blotting or by squeezing the mixed mass in an amalgam squeeze cloth. Patients were recalled and observed periodically during the nine-month observation period.

qq Instron Engineering Corp., Canton, Mass.

#### Results

The properties of the cements evaluated are given in Tables 2-9. The estimated coefficient of variation for the tensile strength values varied widely, but was generally less than 20 percent (maximum value 22.7 percent); compressive strength values gave a maximum coefficient of variation of 14 percent, but were usually less than 10 percent.

The physical properties of commercial ZOE and zinc phosphate cements, the  $Al_2O_3$  reinforced cement reported in our previous study<sup>5</sup> and two methyl methacrylate polymer-containing ZOE type cements that have recently become commercially available are given in Table 2. The EBA and ZOE reinforced crown and bridge cements exhibited improved compressive and tensile strengths when compared to conventional ZOE cements. A marked increase was noted in tensile strength and solubility on mixing the polymer reinforced powder of commercial cement A with a liquid containing 62.5 percent EBA - 37.5 percent eugenol instead of the liquid supplied in the commercial formulation.

Variations in rosins, and to some extent also in zinc oxides, affected the characteristics of the cements (Table 3). An increase in the powder-'liquid ratio of the mix resulted in a decrease in setting time. Tensile strength was decreased when a spherical aluminum oxide was used or when the hydrogenated rosin content was changed from 6 to 8 percent. Using the powder-liquid ratios indicated, the cements containing

rosin were more soluble and set more slowly than those to which hydrogenated rosin had been added.

A maximum tensile strength of 11.5 MN/m<sup>2</sup> (117 kg/sq cm, 1670 psi) at one week

/was obtained on incorporation of a methyl methacrylate copolymer and

rosin (Table 4). Increasing the polymer content of the powder from 9.1 to

13 percent or decreasing it to 4.8 percent lowered the tensile strength

to 9.9 or 8.3 MN/m<sup>2</sup> (101 or 85 kg/sq cm) respectively. Products conusually
taining hydrogenated rosin/presented lower tensile strengths and

markedly lower water solubility and disintegration values, but had higher
compressive strengths than those containing rosin.

Improvements in the physical properties of the formulation containing 58.2 percent ZnO, 27.3 percent Al O, 5.4 percent hydrogenated and 9.1 percent methyl methacrylate copolymer, rosin were not obtained on increasing (1) hydrogenated rosin content the (2) percentage of eugenol or (3) zinc oxide content (and decreasing the percentage of Al O reinforcing agent). Use of a silanized aluminum oxide or a dispersed gamma aluminum oxide in the experimental formulamixing characteristics tions yielded products with inferior transmixes. The latter additive reduced the amount of powder that could be added to the liquid.

Substitution of terpene resin for the rosin ingredient gave a product with a high 24-hour tensile strength that decreased on longer exposure to water because of the high water solubility and disintegration of the product (4.1 percent) in one week. Incorporation of 30 percent

 ${
m Al}_2{
m O}_3$  powders surface coated with varying amounts of polymer in mixes with 64 percent ZnO and 6 percent rosin yielded products having tensile strengths ranging from 6.5 to 7.3 MN/m $^2$  (66 to 74 kg/sq cm) indicating that the coating had little beneficial effect.

The effects of varying (1) particle sizes, (2) the  $\mathrm{CO}_2$ , and (3) water content of the ZnO on the physical properties of the resulting cements were slight (Table 5). Zinc oxide containing only trace amounts of water and  $\mathrm{CO}_2$  and having the smallest particle size yielded products of slightly higher tensile strength. Relatively high percentages of  $\mathrm{CO}_2$  and water in the ZnO decreased the amount of powder that could be incorporated into 0.2 ml of liquid.

Table 6 shows the effects of adding 0.5 percent aluminum sulfate to two of the most promising formulations. A decrease in 1 week water solubility from 0.94 percent to 0.65 percent for rosin-containing cements was obtained when aluminum sulfate was added. There was, however, a decrease in tensile strength and an increase in compressive aluminum strength on incorporating the aluminum salt. The addition of/sulfate also decreased slightly the solubility and disintegration of cements containing hydrogenated rosin.

Table 7 presents the effects of increasing the ethyl methacrylate content of the methyl methacrylate-ethyl methacrylate copolymers used in various formulations. Increasing the ethyl methacrylate content slightly increased the tensile strength. A correlation between resin

composition and compressive strength was not apparent.

Table 8 gives further results of compounding polymers with the powders. Neither the addition of a low molecular weight experimental poly(methyl methacrylate) copolymer (55 D-42) nor a commercial acrylic polymer (KM 228) produced materials with outstanding physical properties. Addition of 9.0 percent or 5.0 percent calcium acrylate to the formulations gave mixes with fast setting times (2.5 and 3.5 minutes). Even faster setting times were obtained with 9.0 percent and 5.0 percent zinc acrylate-(0.5 to 1.5 minutes) and zinc methacrylate-(1 to 2 minutes) containing formulations. Mixes containing resins other than acrylic polymers also hardened readily (Table 9). A vinyl chloride-vinyl acetate copolymercontaining cement had one week tensile and compressive strengths of 9.80  $MN/m^2$  and 91.5  $MN/m^2$  (100 kg/sq cm and 933 kg/sq cm) respectively. Incorporation of an acrylonitrile-butadiene-styrene terpolymer, a polyacetal resin or various grades of commercial polycarbonate molding powders produced materials with physical properties somewhat lower than those containing acrylic copolymer. A much lower tensile strength was obtained with polyvinyl stearate. Addition of a dialdehyde starch gave a cement with a relatively high tensile strength and high solubility.

The clinical study, employing the composition given under Materials and Methods, demonstrated that the cement could be readily adapted to cavity walls and margins. The material did not dissolve or disintegrate in the oral fluids. All restorations remained serviceable and showed only minimal signs of wear over the nine-month observation period. All teeth restored with this cement remained asymptomatic for the entire period of observation.

#### Discussion

The most suitable reinforcing resin for ZOE-EBA cements appear to be methacrylate or possibly vinyl copolymers. Certain other resilient resins may also be potentially useful. These materials, however, because of their resilient nature, cannot be readily obtained in the powdered form and, therefore, were not included in this study.

Since clinical fracture failures of restorations generally occur in tension or shear, high tensile strength values are considered more important than high compressive strengths. The addition of suitable poly mers greatly increases the tensile strengths of some cements. These formulations should last longer under clinical conditions than conventional ZOE products. Clinical studies bear out this point. The formulation selected had a relatively high tensile strength, but the in vitro solubility and disintegration values were considerably higher than those of other promising mixes. Since all restorations remained serviceable over the nine month observation period, in vivo solubility did not appear to be of any significance. Unreacted liquid in the clinical mixes was removed by blotting. It is conceivable that lower in vitro solubility and disintegration values would have been obtained if this procedure had been followed in the preparation of laboratory test specimens.

It would be desirable to evaluate clinically other promising formulations found in this study. Limited clinical evaluation indicates that certain polymer-reinforced materials, on the basis of demonstrated mechanical and palliative properties, appear to be highly useful as long duration temporary restoratives.

#### Conclusions

The effects of the addition of polymeric materials to the powder components of EBA cements were investigated. Acrylic and vinyl copolymers appeared to be the most suitable additives for æhieving reinforcement. The resultant products exhibited significant increases in tensile strength.

These materials present good manipulative qualities. Limited clinical studies have suggested that these materials may be highly desirable for use as long-duration temporary restoratives.

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## Materials

# SOURCES OF POLYMERS

Source

Methyl Methacrylate Copolymers

Acryloid K-120N Acryloid KM228 55D-42

60% Ethyl methacrylate SR-153 P-43-68-385

70% Ethyl methacrylate SR-153 P-45

80% Ethyl methacrylate SR-153 P-45

Poly(ethyl methacrylate) SR-153 P-19-68-39 Poly(ethyl methacrylate) HG-42

Zinc acrylate

Zinc methacrylate

Calcium acrylate

Poly(vinyl chloride 87% - vinyl acetate 13%)

Acrylonitrile-butadiene-styrene terpolymer

Polyacetal

Polycarbonate

Poly(vinyl stearate) Dialdehyde starch

Sartomer Resins, Inc., Essington, Pa. Sartomer Resins, Inc., Essington, Pa. Sartomer Resins, Inc., Essington, Pa. Sartomer Resins, Inc., Essington, Haas, Philadelphia, Pa. Rohm and Haas, Philadelphia, Pa. Rohm and Haas, Philadelphia, Pa. Rohm and

 $\operatorname{Delrin}^R$  100NC10,  $\operatorname{DuPont}$  de Nemours, Wilmington,  $\operatorname{Del}$ . Vinylite VYHH, Union Carbide Co., New York, N. Y. Blender 101 Marbon Chemical, Washington, W. Va. Monomer-Polymer Laboratories, Philadelphia, Pa. Monomer-Polymer Laboratories, Philadelphia, Pa. Sartomer Resins, Inc., Essington, Pa. DuPont de Nemours, Wilmington, Del. Rohm and Haas, Philadelphia, Pa.

Air Reduction Chemical Co., New York, N.Y. Dasol-A Miles Laboratories, Elkhart, Ind. Pittsfield, Mass.

Lexan, 105A, 145, 155, 160 General Electric Co.,

TABLE 2

PHYSICAL PROPERTIES OF COMMERCIAL DENTAL CEMENTS

| Solubility and Disintegration | %                              | 0.02-0.01                | 0.05                   | 0.10-0.20      | 90.0                 | 4.80T                | 0.08                   |
|-------------------------------|--------------------------------|--------------------------|------------------------|----------------|----------------------|----------------------|------------------------|
| ssive<br>gth                  | MN/sq m kg/sq cm               | 140-385                  | 870-930                | 700-1500       | 430                  | 390                  | 770                    |
| Compressive<br>Strength       |                                | 14-38                    | 85-91                  | 69-147         | 42                   | 38                   | 92                     |
| le<br>gth                     | MN <sup>+</sup> /sq m kg/sq cm | 14                       | 67                     | 33-46          | 39                   | 77                   | 70                     |
| Tensile<br>Strength           | W, tsd m                       | 1.4                      | 4.8                    | 3.2-4.5        | 3.8                  | 7.6                  | 6.9                    |
| Setting<br>Time               | min                            | 7-8                      | 8-9                    | 7-8            | 3                    | 2                    | 2                      |
| Powder<br>Liquid<br>Ratio     | gm/m1                          | 1.4/0.4                  | 2.0/0.2                | 1.4/0.5        | 1.1/0.3              | 1.2/0.2              | 1.1/0.2                |
|                               |                                | Zinc Oxide-Eugenol (ZOE) | EBA (Al203 reinforced) | Zinc Phosphate | ZOE Reinforced (B&T) | ZOE Reinforced (B&T) | ZOE Reinforced (Fynal) |

<sup>\*</sup> Liquid: 62.5% EBA - 37.5% Eugenol

<sup>+</sup> MN = Meganewton

 $_{\text{T}}$  One week Solubility and Disintegration

TABLE 3

PROPERTIES OF  $A1_2O_3$  REINFORCED EBA CEMENTS

| y and<br>ation                   | 1 wk<br>%          | I    | ı    | 0.36 | ı    | 0.03  | 0.03 |
|----------------------------------|--------------------|------|------|------|------|-------|------|
| Solubility and<br>Disintegration | 24 hr 1 wk         | 1,   | 1    | 0.13 | 1    | 1     | 1    |
|                                  | C.V.               | ı    | 1    | 3.0  | 2.8  | 0.3   | 8.1  |
| Compressive<br>Strength          | MN/sq m kg/sq cm   | 1    | ı    | 720  | 870  | 1055  | 820  |
| One Week<br>Com<br>St            | m/sd m             | ı    | ı    | 9.07 | 85.3 | 103.5 | 80.4 |
| One                              | C.V.               | 4.8  | 17.3 | 9.6  | 4.7  | 3.3   | 17.6 |
| Tensile<br>Strength              | kg/sq cm           | 09   | 41   | 54   | 26   | 67    | 38   |
|                                  | w bs/NW            | 5.9  | 4.0  | 5.3  | 5.5  | 4.8   | 3.7  |
| Setting<br>Time                  | min                | 11.5 | 1    | 10.5 | 7.5  | 7.0   | 6.5  |
| Powder<br>Liquid<br>Ratio        | gm/0.2 ml          | 1.2  | 1.2  | 1.2  | 1.7  | 2.0   | 2.0  |
|                                  | ·H                 |      |      |      | +.0  | +.0   | T-00 |
| Composition<br>of<br>Powder      | $^{A1}_{2\%3}^{0}$ | 30   | 30+  | 30   | 30   | 30    | 30   |
|                                  | Zn0<br>%           |      |      |      |      |       |      |

Source of ZnO: The New Jersey Zinc Co. Spherical aluminum oxide Hydrogenated rosin

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TABLE 4

PROPERTIES OF ACRYLIC\* REINFORCED EBA RESTORATIVE MATERIALS

| Solubility and<br>Disintegration | 24 hour 1 week $\%$ $\%$       | 1    | 1         | 0.13 0.20 | 1            | 1      | 1          | 0.39 0.94 | 60.4  | ı    | 1    | 1     | ,                           | 1         | 1         |  |                    |
|----------------------------------|--------------------------------|------|-----------|-----------|--------------|--------|------------|-----------|-------|------|------|-------|-----------------------------|-----------|-----------|--|--------------------|
|                                  | c.v.                           | 1    | ı         | 8.9       | 6.1          | 13.4   | ı          | 9.4       | 9.6   | 1    | ,    |       | 1                           | ı         | ı         |  |                    |
|                                  | week<br>kg/sq cm               | 1    | ı         | 821       | 550          | 417    |            | 799       | 480   | ,    | 1    | 1     | ı                           |           |           |  |                    |
| .ve<br>.h                        | 1<br>MN/sq m                   | ı    | ı         | 80.5      | 53.9         | 6.04   | ı          | 65.1      | 47.1  | 1    | 1    | 1     | ı                           | •         | 1         |  |                    |
| Compressive<br>Strength          | cm %                           | ı    | 15.8      | ı         | 1            | ı      | ı          | 11.1      | 7.8   | 6.0  | 1    | ı     | ı                           | 5.5       | 8.8       |  |                    |
| 0                                |                                | ı    | 641       | ı         | ı            | 1      | ı          | 514       | 364   | 592  | 1    | 1     | •                           | 029       | 629       |  |                    |
|                                  | 24 hour<br>MN/sq m kg/sq       | 1    | 62.9      | 1         | •            | ı      | 1          | 50.4      | 35.7  | 58.1 | 1    | ı     | 1                           | 65.7      | 9.49      |  |                    |
|                                  | c.v.                           | 5.4  |           | 9.4       | 11.1         | 8.7    | 9.9        | 7.1       | 20.7  | ı    | 10.4 | 12.9  |                             | ı         | ı         |  |                    |
|                                  | 1 week<br>m kg/sq cm           | 101  |           | 7.5       | 96           | 78     | 87         | 117       | 79    | ı    | 85   | 63    | ,                           | ı         | 1         |  |                    |
| ्री                              | 1 v<br>NN/sq m Þ               | 6.6  | 1         | 7.4       | 9.3          | 7.6    | 8.5        | 11.5      | 6.3   | ı    | 8.3  | 6.2   | 1                           | ı         | •         |  | ide                |
| Tensile<br>Strength              | C.V.                           | ı    | 22.7      | 10.4      | ı            | 1      | ı          | 3.8       | 6.5   | 14.1 | ı    | 1     | ,                           | 10.6      | 14.1      |  | aluminum oxide     |
|                                  | ur<br>g/sq cm                  | ı    | 89        | 7.4       | 1            | 1      | ı          | 66        | 101   | 63   | 1    | ı     | ı                           | 99        | 62        | )3   | mma alum           |
|                                  | 24 hour<br>M/sq m kg/sq        | ı    | 6.7       | 7.3       | ı            | ı      | 1          | 6.7       | 6.6   | 6.2  | ı    | 1     | ,                           | 6.3       | 6.1       | 0.5% Al <sub>2</sub> (SO <sub>2</sub> ) <sub>3</sub> | ++ Dispersed gamma |
| Setting<br>Time                  | min                            | 7    | 1         | 7.5       | 8.5          | 1      | 10.5       | 7.0       | 5.0   | 1    | 6    | 6.5   | 2                           | 1         | ,         | ** 0.5   | + Dis              |
| Powder-<br>Liquid                | g/0.2 ml                       | 1.1  | 1.2       | 1.3       | 1.1          | 1.1    | 1.1        | 1.1       | 1.3   | 1.4# | 1.1  | 1.1   | 0.5                         | 1.4       | 1.7       |  |                    |
|                                  | Poly-<br>mer g                 | 13.0 | 9.1       | 9.1       | 9.1          | 9.1    | 9.1        | 9.1       | 9.1   | 9.1  | 4.8  | 8°0°6 | 9.1                         | 9.1       | ı         |  |                    |
| Composition<br>of<br>Powder      | Hydr.<br>Rosin<br>%            | 5.2+ | 7.3       | 5.4       | 5.4          | 5.4+   | 5.4+       | 5.4+      | 5.4 € | 5.4  | 5.7+ | 5.3   | 5.4                         | 5.4       | 0.9       | K-120N   |                    |
| Сотрс                            | $^{2n0}_{\%}$ A1 $^{0}_{2\%3}$ | 26.1 | 56.3 27.3 | 58.2 27.3 | 58.2 \$ 27.3 | 27.3 ™ | 58.28 27.3 | 58.2 27.3 | 27.3  | 27.3 | 28.6 | 20.0  | 65.5 20.0 <sup>++</sup> 5.4 | 71.8 13.6 | 79.0 15.0 | * Acryloid K-120N                                    | osin               |
|                                  | %<br>0uZ                       | 55.7 | 56.3      | 58.2      | 58.2         | 58.2   | 58.2       | 58.2      | 58.2  | 58.2 | 6.09 | 65.2  | 65.5                        | 71.8      | 79.0      | *  | + Rosin            |

<sup>+</sup> Rosin
S N. J. Zinc Co.

m Silanized

<sup>¶</sup> Terpene resin

<sup>#</sup> Liquid: 50% EBA-50% eugenol

TABLE 5

EFFECT OF PARTICLE SIZE, WATER AND CARBON DIOXIDE CONTENT OF ZINC OXIDES ON THE PROPERTIES OF REINFORCED EBA RESTORATIVE MATERIALS

\*Powder: 58.2% ZnO, 27.3% Al<sub>2</sub>O<sub>3</sub>, 5.4% Rosin, 9.1% Copolymer

| gth  | C.V.    | %          | 8.0  | 3.4  | 7.9       | 4.5     | 7.6     | 8.3     |
|--|---------|------------|------|------|-----------|---------|---------|---------|
| Compressive Strength                         |         | kg/sq cm % | 720  | 740  | 069       | 639     | 662     | 634     |
| Compressi                                    |         | MN/sd m    | 70.6 | 72.6 | 67.7      | 62.7    | 6.49    | 62.2    |
|  | C • V • |            | 1.8  | 15.5 | 9.8       | 9.2     | 14.0    | 6.9     |
| Tensile Strength                             |         | kg/sq cm   | 6    | 89   | 82        | 83      | 06      | 86      |
| Tensil                                       |         | WN/sd m    | 9.5  | 8.7  | 8.0       | 8.1     | 8.8     | 8.4     |
| Powder<br>Liquid                             | Ratio   | gm/0.2 ml  | 1.1  | 1.1  | 1.1       | 8.0     | 1.0     | 1.0     |
| Н,0  | 1       | %          | ni1  | ni1  | 0.37 0.38 | 3.8     | 1.5     | 1.4     |
| $2n0^{+}$                                    | 1       | %          | nil  | ni1  | 0.37      | 4.8 3.8 | 1.8 1.5 | 1.6 1.4 |
| $2n0^+$ Powder Particle $CO_2$ $H_2O$ Liquid | Size    | шn         | 0.37 | 0.57 | 0.37      | 0.37    | 0.57    | 0.92    |

\* Acryloid K-120N + N. J. Zinc Co.

EFFECT OF ADDITION OF ALUMINUM SULFATE ON THE PROPERTIES OF PLASTIC REINFORCED EBA RESTORATIVE MATERIALS

Powder: 58.2% ZnO, 27.3%  ${\rm Al_2}^{0}_3$ , 5.4% Rosin, 9.1% Polymer

| y and                                     | 1 week  | 0.94          | 0.65 | 0.20 | 0.13     |
|---|---|---------------|------|------|----------|
| Solubility and<br>Disintegration          | 24 hour %   | 4.6 0.39      | 0.39 | 0.13 | 0.09     |
| 63 11                                     | C.V.  | 9.4           | 2.0  | 8.9  | 4.4      |
|   | ek<br>g/sq cm   | 799           | 874  | 821  | 786      |
| rength                                    | l week c.v. 24 hour c.v. 1 week c.v. 24 hour 1 week MN/sq m kg/sq cm $\%$ MN/sq m kg/sq cm $\%$ MN/sq m kg/sq cm $\%$ % | 65.1 664      |      | 80.5 | 77.1 786 |
| ive St                                    | C.V.  | 11.1          | ı    | ı    | 1        |
| Compressive Strength                      | t hour<br>g/sq cm   | 50.4 514 11.1 | ı    | ı    | 1        |
| 0   | 27<br>WM/sq m 1   | 50.4          |      | I    | 1        |
|   | c.v.  | 7.1           | 9.9  | 9.4  | 8.4      |
| -   | week<br>kg/sq cı  | 11.5 117 7.1  | 102  | 75   | 74       |
| Tensile Strength                          | 1<br>MN/sq m 1  | 11.5          |      | 7.4  | 7.4      |
| Tensil                                    | c.v.<br>m %   | 3.8           | 4.8  | 10.4 | 14.8     |
|   | hour<br>8/sq c  | 66            | 06   | 74   | 99       |
|   | 24 hour c.v. MN/sq m kg/sq cm %   | 6.7           | 8.8  | 7.3  | 6.5      |
| Setting<br>Time                           | min   | 7             | 7.5  | 7.5  | ∞        |
| Powder<br>Liquid<br>Ratio                 | gm/0.2 ml   | 1.1           | 1.2  | 1.3  | 1.2      |
| $\mathrm{Al}_2(\mathrm{SO}_4)_3$<br>Added | %   |               |      |      |          |

\* Acryloid - K-120N

+ Hydrogenated rosin  $\delta$  Composition used in clinical evaluation

TABLE 7

EFFECTS OF COPOLYMER COMPOSITION ON THE PROPERTIES OF REINFORCED EBA RESTORATIVE MATERIALS

Powder: 58.2% ZnO, 27.3%  $\mathrm{Al}_2\mathrm{O}_3$ , 5.4% Hydrogenated Rosin, 9.1% Acrylic Copolymer

|                                  | C . V .      | %                      | 1       | 13.6 | 4.0  | 13.5 | 1.2  | 1    | 14.0        |
|----------------------------------|--------------|------------------------|---------|------|------|------|------|------|-------------|
|                                  | l week o     | g/sq cm                |         | 579  | 892  | 735  | 840  | 1    | 728         |
| Compressive<br>Strength          | I w          | MN/sq m kg/sq cm       | 1       | 56.8 | 87.5 | 72.1 | 82.4 | 1    | 71.3        |
|                                  | C.V.         | %<br>"                 | 60 10.8 | 15.9 | 22.0 | 20.3 | 7.8  | 18.5 | 21.0        |
| le<br>gth                        | ek           | s/sd cn                | 09      | 41   | 36   | 47   | 58   | 73   | 44          |
| Tensile<br>Strength              | 1 week       | MN/sq m kg/sq cm %     | 5.9     | 0.4  | 3.5  | 4.5  | 5.7  | 7.2  | 4.3         |
| Setting<br>Time                  |              | min                    | 10.5    | 6.5  | 6.5  | 7.0  | 7.0  | 1    | 7.0         |
| i m                              | Ratio        | gm/0.2 ml              | 1.2     | 1.8  | 2.0  | 2.0  | 2.0  | 1.6  | 2.0         |
| omposition                       | ylate        | Ethyl<br>%             |         | 09   | 70   | 80   | 100  | 100+ | <del></del> |
| <pre>Copolymer Composition</pre> | Methacrylate | $\texttt{Methy1}\\ \%$ | SR-120  | 40   | 30   | 20   | 1    | 1    | P43-68-385  |

<sup>\*</sup> Sartomer Resin

<sup>+</sup> DuPont HG42 and 5.4% rosin

<sup>†</sup> Copolymer composition not given by manufacturer

TABLE 8

PROPERTIES OF ACRYLIC REINFORCED EBA RESTORATIVE MATERIALS

|                                   | C.V.   | 11.7        | ı        | ı         | i         |  |
|-----------------------------------|--|-------------|----------|-----------|-----------|--|
|                                   | k<br>g/sq c  | 512         | 1        | ı         | ŧ         |  |
| ıgth                              | c.v. 1 week<br>m % MN/sq m kg/                                     | 50.2 512    | 1        | 1         | ı         |  |
| Compressive Strength              | c.v.<br>n % N  | 355 9.0     | 490 12.4 | ı         | ı         |  |
| ressive                           | ır<br>g/sq cı  |             | 760      | ı         | ı         |  |
| Сомрт                             | 24 hour c.v. 1 week c.v. $MN/sq$ m kg/sq cm % $MN/sq$ m kg/sq cm % | 34.8        | 48.1     | 1         | 1         |  |
|                                   |  | 10.2        | ı        | 14.0      | 12.7      |  |
|                                   | 1 week c.v.<br>m kg/sq cm  | 79 10.2     | 1        | 69        | 7.1       |  |
| ength                             | 1<br>M/sq m  | 7.7         | ı        | 8.9       | 7.0       |  |
| Tensile Strength                  | c.v.   | 63 14.1 7.7 | 75 15.6  | ı         | ı         |  |
| Tensi                             | hour<br>kg/sq c  | 63          | 7.5      | ı         | ı         |  |
|                                   | 24<br>MN/sq m  | 6.2         | 7.4      | ı         | 1         |  |
| Powder<br>Liquid                  | Ratio<br>gm/0.2 ml   | 1.2         | 1.8      | 1.8       | 1.8       |  |
| omposition of<br>Powder<br>Ivdr.  | Rosin Polymer % g  | 5.4 9.1*    | 48.8     | 3.8+      | 2.0+      |  |
| Composition of<br>Powder<br>Hydr. | Rosin<br>%   | 5.4         | 5.7      | 5.8+      | 5.9+      |  |
|                                   | $^{ m A1}_{2}{}^{ m 0}_{\rm 3}$                                    | 27.3        | 28.6     |           | 29.4      |  |
|                                   | ZnO<br>%   | 58.2 27.3   | 6.09     | 61.5 28.9 | 62.7 29.4 |  |

<sup>\* 55</sup>D-42-Rohm and Haas

<sup>+</sup> Rosin

<sup>†</sup> KM 228-Rohm and Haas

TABLE 9

PROPERTIES OF PLASTIC REINFORCED EBA RESTORATIVE MATERIALS

Powder: 58.2% ZnO, 27.3%  $\mathrm{Al_2}^{0}$ 3, 5.4% Hydrogenated Rosin, 9.1% Polymer

| Polymer                             | Powder<br>Liquid<br>Ratio | Setting<br>Time |                  | Tensile<br>Strength<br>1 week | D.   | Compressive<br>Strength<br>1 week | ssive<br>gth<br>ek | C . V | Solubility and<br>Disintegration<br>24 hour l week | y and<br>ation<br>week |
|-------------------------------------|---------------------------|-----------------|------------------|-------------------------------|------|-----------------------------------|--------------------|-------|--|------------------------|
|                                     | g/0.2 ml                  | min             | MN/sq m kg/sq cm | o bs/8s                       |      | MN/sq m kg/sq cm %                | g/sq cn            | 2 %   | %  | %                      |
| Vinyl chloride (87%) vinyl          | 1.3                       | 11              | 8.6              | 100                           | 10.0 | 91.5                              | 933                | 3.0   | 0.17   | 0.55                   |
| acerare (13%)<br>Polyvinyl stearate | 2.0                       | 6.5             | 2.9              | 30                            | 8.8  | ı                                 | ı                  | 8     | ı  | ı                      |
| ABS-Terpolymer                      | 1.8                       | 6.5             | 5.6              | 57                            | 5.8  | 48.7                              | 467                | 0.6   | ı  | ı                      |
| Polyacetal                          | 2.0                       | 6.5             | 5.1              | 52                            | 8.6  | 4.89                              | 269                | 1.9   | ı  | ı                      |
| Lexan 105A                          | 1.6                       | 7.5             | 5.7              | 58                            | 13.0 | 78.1                              | 962                | 1.7   | ı  | ı                      |
| Lexan 145                           | 1.3                       | 8               | 5.7              | 58                            | 10.7 | 55.4                              | 565                | 10.0  | ı  | 1                      |
| Lexan 145*                          | 1.2                       | 6               | 6.2              | 63                            | 11.4 | ı                                 | ı                  | ı     | 1  | ı                      |
| Lexan 155                           | 1.6                       | 8               | 5.6              | 57                            | 8.9  | 45.5                              | 494                | 2.8   | ı  | ı                      |
| Lexan 160                           | 1.6                       | 7.5             | 5.8              | 59                            | 5.8  | 53.3                              | 544                | 5.7   | 1  | ı                      |
| Dialdehyde starch*                  | 1.2                       | 1               | 8.9              | 91                            | 3.6  | ı                                 | ı                  | ı     | 3.72   | 1                      |
|                                     |                           |                 |                  |                               |      |                                   |                    |       |  |                        |

<sup>\* 5.4%</sup> Rosin



